

5-Chlorocarbonyl-10,11-dihydro-5H-dibenz[*b,f*]azepineT. Vijay,<sup>a</sup> H. G. Anilkumar,<sup>b</sup>  
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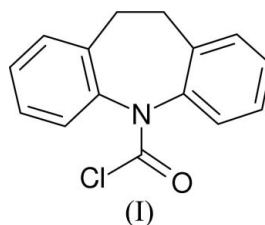
## Key indicators

Single-crystal X-ray study  
 $T = 295\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$   
 $R$  factor = 0.063  
 $wR$  factor = 0.119  
Data-to-parameter ratio = 17.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound,  $\text{C}_{15}\text{H}_{12}\text{ClNO}$ , the central seven-membered azepine ring adopts a bent conformation, intermediate between the boat and chair forms. The overall structure of the molecule is similar to a butterfly shape, which is commonly observed for carbamazepine analogues. The planes through the benzene rings on either side of the azepine ring intersect at an angle of  $59.0(1)^\circ$ . The molecular assembly is primarily stabilized by aromatic  $\pi$ - $\pi$  interactions.

## Comment

Carbamazepine and its analogues, which belong to the family of iminostilbenes, have been widely used therapeutically as anticonvulsant agents (Pearce *et al.*, 2002; Czapinski *et al.*, 2005). In order to study the structure–activity relationships of these compounds, numerous structures of iminostilbene derivatives have been reported (Lisgarten *et al.*, 1989; Lang *et al.*, 2002; Hempel *et al.*, 2005; Nagaraj *et al.*, 2005; Johnston *et al.*, 2005). The crystal structure analysis of the title compound, (I), reported here was undertaken as a part of an ongoing programme of structural studies of molecules of pharmaceutical interest.



The molecular structure and conformation of (I) are shown in Fig. 1. The planes of atoms  $\text{N5}/\text{C1}-\text{C4}/\text{C11}-\text{C13}$  and  $\text{N5}/\text{C6}-\text{C9}/\text{C10}/\text{C11}/\text{C14}/\text{C15}$  intersect at an angle of  $59.0(1)^\circ$ . The central seven-membered azepine ring ( $\text{N5}/\text{C10}-\text{C15}$ ) adopts a bent conformation with five coplanar atoms ( $\text{N5}/\text{C10}/\text{C11}/\text{C14}/\text{C15}$ ) (Bocian *et al.*, 1975; Bocian & Strauss, 1977). Six distinct conformations have been identified and characterized for a classical cycloheptane ring: (i) boat, (ii) chair, (iii) twist-boat, (iv) twist-chair, and the two high-energy intermediate transition states, namely, (v) bent transition state between the first two and, (vi) twisted transition state between the latter two (Hendrickson, 1961, 1967; Bocian *et al.*, 1975; Bocian & Strauss, 1977). The bent transition state of the seven-membered ring with five coplanar atoms is one of the most popular conformation among carbamazepine analogues, which has been improperly referred to as twist-boat or half-boat by many authors. In addition to the puckering parameters of the ring described in the literature (Bocian *et al.*, 1975; Bocian & Strauss, 1977), the 'twist' nomenclature in its classical sense

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mean the form with  $C_2$  (axial) symmetry (Hendrickson, 1961, 1967), while the present conformation of the ring possesses  $C_s$  (mirror plane) symmetry. The puckering parameters (Cremer & Pople, 1975) of the azepine ring are,  $q_2 = 0.813$  (3) Å,  $q_3 = 0.284$  (3) Å,  $\varphi_2 = 202.9$  (2)° and  $\varphi_3 = 125.4$  (7)°, and the total puckering amplitude  $Q_T = 0.861$  (3) Å. The internal torsion angles of the azepine ring are indicated in Fig. 1. The bent conformation of the seven-membered ring possesses a mirror symmetry about the plane passing through atom C15 and bisecting the C12–C13 bond. The asymmetry parameter (Duax *et al.*, 1976),  $\Delta C_s(m)$ , is 8.2°. Due to the present conformation of the azepine ring, the molecule assumes a butterfly shape, as previously observed (Hempel *et al.*, 2005; Nagaraj *et al.*, 2005).

In the absence of any potential hydrogen-bond donors, the crystal packing of (I) is mainly stabilized by aromatic  $\pi$ – $\pi$  interactions of the edge-to-face type, in addition to van der Waals forces (Fig. 2). The geometric parameters, *i.e.* centroid···centroid separations, interplanar angles and minimal C–C separations in the  $\pi$ – $\pi$  interactions, respectively, are as follows  $Cg1 \cdots Cg1(2-x, 2-y, -\frac{1}{2}+z) = 4.926$  (2) Å, 32.3°, 3.46 Å;  $Cg1 \cdots Cg2(2-x, 1-y, \frac{1}{2}+z) = 5.057$  (2) Å, 79.0°, 3.60 Å;  $Cg2 \cdots Cg2(\frac{3}{2}-x, y, -\frac{1}{2}+z) = 4.987$  (2) Å, 70.5°, 3.76 Å.  $Cg1$  and  $Cg2$  are the centroids of ring 1 (atoms C1–C4/C12/C13) and ring 2 (atoms C6–C9/C14/C15), respectively.

## Experimental

The title compound (Jubilant Organosys, Nanjangaud, India) was crystallized from ethyl methyl ketone.

### Crystal data

$C_{15}H_{12}ClNO$   
 $M_r = 257.71$   
 Orthorhombic,  $Pca2_1$   
 $a = 16.654$  (3) Å  
 $b = 10.646$  (2) Å  
 $c = 7.2599$  (14) Å  
 $V = 1287.1$  (4) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.330$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 870 reflections  
 $\theta = 5$ – $27^\circ$   
 $\mu = 0.28$  mm<sup>-1</sup>  
 $T = 295$  (2) K  
 Plate, colourless  
 $0.58 \times 0.20 \times 0.12$  mm

### Data collection

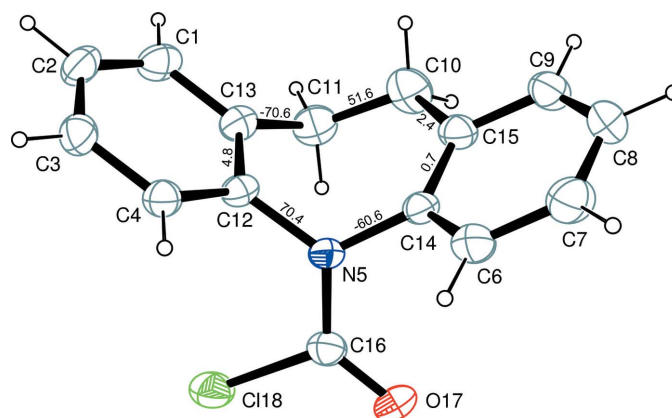
Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.926$ ,  $T_{\max} = 0.971$   
 6801 measured reflections

2890 independent reflections  
 2159 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.032$   
 $\theta_{\text{max}} = 27.9^\circ$   
 $h = -18 \rightarrow 21$   
 $k = -14 \rightarrow 11$   
 $l = -9 \rightarrow 8$

### Refinement

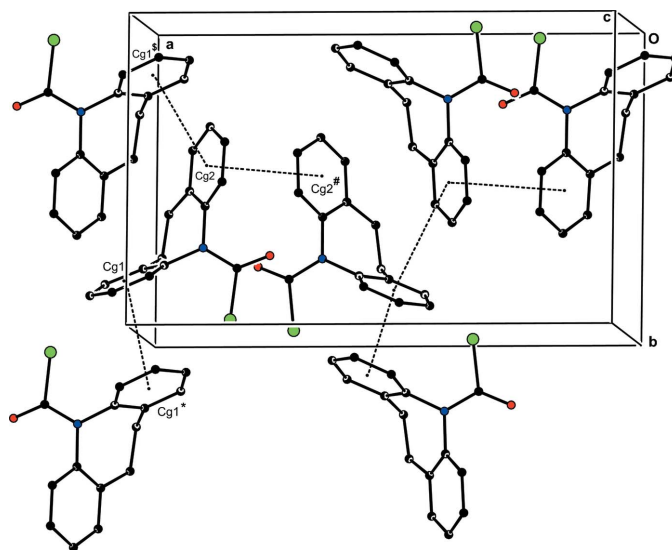
Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.063$   
 $wR(F^2) = 0.119$   
 $S = 1.13$   
 2890 reflections  
 163 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.0447P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.29$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983), with 1414 Friedel pairs  
 Flack parameter: 0.03 (10)



**Figure 1**

A view of molecule (I). The numerical figures refer to the internal torsion angles (°) of the central azepine ring in a bent conformation; s.u. values are in the range 0.4–0.5°. Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as small spheres of arbitrary radii.



**Figure 2**

The crystal packing of (I), stabilized by  $\pi$ – $\pi$  interactions.  $Cg1$  and  $Cg2$  are the centroids of ring 1 and ring 2, respectively. Atoms labelled with an asterisk (\*), dollar sign (\$) or hash (#) are at the symmetry positions  $(2-x, 2-y, -\frac{1}{2}+z)$ ,  $(2-x, 1-y, -\frac{1}{2}+z)$  and  $(\frac{3}{2}-x, y, -\frac{1}{2}+z)$ , respectively. Colour key: C black, H white, Cl green, N blue, O red.

All H atoms were positioned geometrically and refined as riding on their parent atoms, with  $C_{\text{ar}}\text{--H} = 0.93$  Å and methylene  $C\text{--H} = 0.97$  Å, and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: SMART (Bruker, 2001); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PLATON.

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